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PATENT

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of:	Tokas, et al.	Docket No.:	IR-2588(ET)
Serial No.:	09/209,706	Group Art Unit:	1733
Filed :	December 11, 1998	Examiner:	G. Knable
For:	*"Contact Metathesis Polymerization"*		

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Assistant Commissioner for Patents
Washington, DC 20231

DECLARATION PER 37 C.F.R. 1.132

Sir:

This is in response to the Office Action dated Oct. 23, 2002. The purpose of this declaration is to provide a showing of unexpected results in support of the patentability of the claimed invention over U.S. Pat. No 5,491,206 to Brown-Wensley et al.

The undersigned Applicant declares the following:

As a below named inventor, we hereby declare that: My residence, post office address and citizenship is as stated below next to my name;

I further declare the following:

1. Comparative coating examples on metal and Teflon® coated panel substrates were conducted to compare the techniques taught in Brown-Wensley, et al to the present invention. A representative example was chosen from Example 18 of the Brown-Wensley Patent, noted at Col. 18, beginning on line 65.

2. Example 18 calls for an iridium catalyst. However this type of catalyst is not readily available. In its place, I used an equally effective metathesis catalyst known as Grubbs' 1st generation catalyst [$RuCl_2(PCy_3)_2$=CHPh] in each of the examples. This catalyst is representative of a primary catalyst described in the instant patent application.

3. The monomer levels and concentrations of catalyst and monomer used in the comparative tests below were based on the same monomer to catalyst level used in Example 18 of Brown-Wensley.

4. A silane promoter was eliminated for each comparative because such a promotor is not required with a ruthenium catalyst.

5. The weight amounts of dicyclopentadiene and CH_2Cl_2 were the same as followed in Example 18 of Brown-Wensley.

6. Preliminary coating runs were performed to develop a feasible facsimile to match example 18 as close as possible. As a summary, a concentrated solution of Grubbs' catalyst was added to dicyclopentadiene in CH_2Cl_2 and stirred. After about 10-15 seconds, the mixture began to thicken. This mixture was then poured onto a 3"x 4" steel panel that was previously wiped with isopropanol and air-dried. In one example, a Teflon panel was used. After coating the panels, and waiting about 5 minutes, the panels were placed into an oven at 100 °C for 5 minutes, then cooled to room temperature in about 5 minutes, and examined for adhesion by cross-hatch testing.

7. The preliminary coatings were attempted. The coating solutions underwent rapid thickening on mixing catalyst, solvent and monomer but coating was attempted on Teflon coated plaque and steel plaques. The coated mass of polymer was readily pulled up from the Teflon surface as the sample cooled. Attempts were made to pour the thick liquid onto a steel surface. The coating thickness on the steel panels was less than on the Teflon surface, but was too thick for meaningful testing (See Table belew, Steel 1).

8. In the second set of experiments, the same method was used except coatings were made within about 10 seconds of mixing before the viscosity level rose as high as in the preliminary runs. An aliquot of about 50 wt% of the solution mixture was cast onto a steel panel and allowed to flow out onto the surface. The remaining solution was cast onto another steel panel and spread over the surface using a raised glass rod to provide a coating . The two coatings were nearly the same thickness, but the one that was poured (See Table, Steel 2) was les uniform than that of film prepared by spreading with the glass rod (Table, Steel 3). Both coated plates were allowed to stand for approximately 5 minutes before placing into the oven.

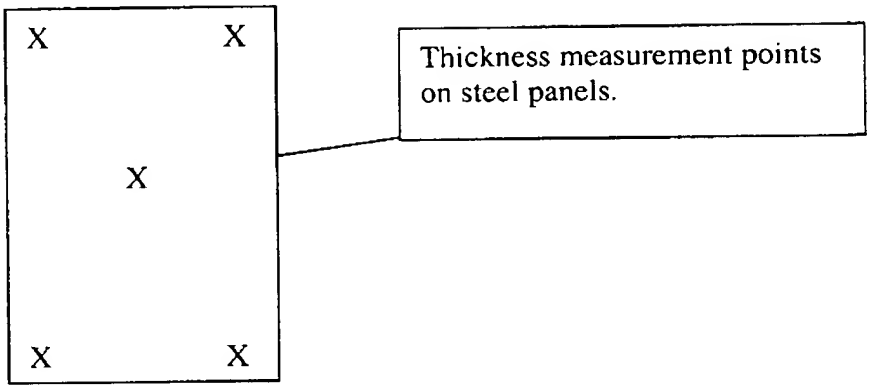
9. After heating and cooling, both coated plaques, the films showed signs of poor adhesion to the steel surfaces as evidenced by pocket formation underneath the films.

10. Cross-hatch adhesion of all samples using scored lines and Scotch tape, revealed the films cast according to U.S. Pat. No 5,491,206 to Brown-Wensley et al. to have cohesive strength, however adhesion to the surface was poor on both Teflon® and steel panels as evidenced by the entire film being lifted from the surface during the Scotch tape peel. The film on one coated plaque could also be separated from the panel surface by lifting an edge of the film with a spatula.

TABLE - Examples according to U.S. Pat. No 5,491,206 to Brown-Wensley et al.

Thickness Measurements (mm)	Teflon 1	Steel 1	Steel 2	Steel 3
1	-	1.73	0.46	0.42
2	-	1.80	0.45	0.43
3	-	1.97	0.34	0.41
4	-	1.64	0.20	0.48
5	-	1.41	0.20	0.39
Ave	-	1.71	0.33	0.43
Stdev	-	0.21	0.13	0.03

Coupon plaque



Part 2.

Repeat Experiment Side – By – Side comparison

Polydicyclopentadiene coatings were prepared on steel using the premix method according to the prior art, and the contact metathesis method according to the invention using the same steel substrates and having dimensions of 4" x 3" x 1/16" and believed to be 1006/1020 carbon steel coupons. The steel surfaces were first prepared by wiping with a rag soaked with isopropanol.

Film thickness was measured using a Veeco MP-900 (Veeco Instruments) film thickness gauge.

Cross-hatch adhesion tests were performed by using a razor blade to cut a 5 x 5 grid through the coating. The cross-hatched area was then covered with a piece of Scotch

232 (3M) masking tape. A wooded tongue depressor was used to press down the tape. The tape was then peeled off in one rapid, smooth motion. Percent adhesion was calculated by subtracting from one the ratio of the removed squares (whole and fractional) to the total number of squares and multiplying by 100.

Contact metathesis method

In the contact metathesis method according to the invention, a solution of 25 mg (0.030 mmol) of Grubbs' first generation catalyst was dissolved in 15 mL of dichloromethane. This solution was placed into a Badger air-brush paint sprayer (Badger Air-Brush Company) attached to Badger Propel spray paint propellant. The catalyst solution was sprayed onto 1.5" x 1.5" areas on two steel coupons. The solvent was allowed to evaporate for 30 minutes.

A solution of 5.51 g (41.7 mmol) of dicyclopentadiene in 4.6 g of dichloromethane was prepared, filtered through a 0.45 micron PTFE filter, and placed in the air-brush sprayer. For one sample, a single pass of the dicylopentadiene was sprayed over the catalyst area. For the second sample two passes were made, allowing approximately 5 minutes between passes for the monomer to react.

These procedures were repeated on two more 1.5" x 1.5" areas of the steel coupons, with the exception that three and four passes of the monomer solution were used. The films were allowed to dry at room temperature for 24 hours.

Premix method according to the prior art

The premix method according to the prior art was followed by pouring a premixture of monomer and catalyst solution onto the steel panels. The mixtures were prepared by dissolving 5.51 g (41.7 mmol) of dicyclopentadiene in 4.6 g of dichloromethane and filtering through a 0.45 micron PTFE filter. A solution of 25 mg (0.030 mmol) of Grubbs' first generation catalyst dissolved in 0.5 mL of dichloromethane was added to the monomer solution.

The reacting mixtures were rapidly mixed and then poured onto the steel coupons. A 1 mil wire-wound draw-down bar was used to prepare an even film. A second film was prepared from the same reacting mixture. The films were allowed to cure at room temperature for 5 minutes and then at 100 °C for 5 minutes. The samples were removed from the oven and allowed to cure for an additional 24 hours at room temperature. The procedure was repeated to make a single film with a 2 mil draw-down bar.

The results of the film-thickness and adhesion tests are tabulated below.

Invention			Premix method		
Passes	Film Thickness	Adhesion	Pull-down bar	Film Thickness	Adhesion
1	0.33 mil	100%	1 mil	0.5 mil	0%
2	0.61 mil	94%	1 mil	0.5 mil	0%
3	0.92 mil	26%	2 mil	0.57 mil	56%
4	1.05 mil	94%			

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

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CERTIFICATE OF MAILING (37 CFR 1.8(A))

I hereby certify that this paper (along with any paper referred to as being attached or enclosed) is being deposited with the United States Postal Service on November 20, 2002 with sufficient postage as first class mail in an envelope addressed to the Assistant Commissioner of Patents, Washington, DC 20231.

Alida M. Clark

February 24, 2003
Date